ORGIA INSTITUTE OF TECHNOLOGY | OFFICE OF CONTRACT ADMINISTRATION

PROJECT ADMINISTRATION DATA SHEET

[x] ORIGINAL [ ] REVISION NO. ______

oject No. A-3337 DATE 9/13/82

oject Director: J. N. Harris ~~School~~/Lab EMSL

onsor: Energy & Minerals Research Company

pe Agreement: Agreement dated 8/24/82 under Contract No. N60921-82-C-0125

ward Period: From 8/24/82 To ~~11/24/82~~ 1/24/83 (Performance) 1-31-83 (Reports)

onsor Amount: $4,452 (NTE) Contracted through:

st Sharing: NA GTRI/~~GIT~~

tle: Support of Ultrasonically Assisted Slip-casting of Fused Silica

DMINISTRATIVE DATA

OCA Contact Linda H. Bowman x4820

Sponsor Technical Contact:

2) Sponsor Admin/Contractual Matters:

Mr. Ben A. Valocchi
VP Administration & Treasurer
Energy & Minerals Research Co.
P. O. Box 389
964 E. Swedesford Road
Exton, PA 19341

fense Priority Rating: NA

Security Classification: NA

ESTRICTIONS

e Attached NA Supplemental Information Sheet for Additional Requirements.

avel: Foreign travel must have prior approval — Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

uipment: Title vests with Gov't prime; however, none proposed

OMMENTS:

SEP 1982 RECEIVED Research Reports Office

OPIES TO:

dministrative Coordinator	Research Security Services	EES Public Relations (2)
esearch Property Management	Reports Coordinator (OCA)	Computer Input
ccounting	Legal Services (OCA)	Project File
ocurement/EES Supply Services	Library	Other ______

RM OCA 4:781

3R 55 R

GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION SHEET

Date 3/1/83

Project Title: Support of Ultrasonically Assisted Slip-Casting of Fused Silica

Project No: A-3337

Project Director: Dr. J. N. Harris

Sponsor: Energy & Minerals Research Co.

Effective Termination Date: 1/24/83

Clearance of Accounting Charges: 1/31/83

Grant/Contract Closeout Actions Remaining:

- [x] Final Invoice and Closing Documents
- [ ] Final Fiscal Report
- [x] Final Report of Inventions
- [x] Govt. Property Inventory & Related Certificate
- [ ] Classified Material Certificate
- [ ] Other ______

Assigned to: EMSL (School/Laboratory)

COPIES TO:

Administrative Coordinator
Research Property Management
Accounting
Procurement/EES Supply Services
Research Security Services
Reports Coordinator (OCA)
Legal Services (OCA)
Library
EES Public Relations (2)
Computer Input
Project File
Other Proj Dir.

FORM OCA 10:781

A-3337

ENGINEERING EXPERIMENT STATION
Georgia Institute of Technology
A Unit of the University System of Georgia
Atlanta, Georgia 30332

February 2, 1983

Energy and Minerals Research Company
P. O. Box 389
964 E. Swedesford Road
Exton, Pennsylvania 19341

Attention: Dr. Scott Taylor

Subject: Final Report, "Support of Ultrasonically Assisted Slip Casting of Fused Silica," Agreement dated 082482 under Contract No. N60921-81-C-0125, Georgia Tech Project A-3337

Gentlemen:

This report describes the three tasks conducted for the Energy and Minerals Research Company in support of their effort on ultrasonic assisted slip casting of fused silica. These three tasks are:

(1) Preparation of a commercial high purity fused silica slip and the conventional casting of fused silica plates therefrom.

(2) Preparation of high purity fused silica grain to be used in aggregate-slip casting operations.

(3) Sintering of conventionally slip-cast and ultrasonically assisted slip-cast fused silica plates.

Task 1

A. Preparation of Commercial High Purity Fused Silica Slips

The Energy and Minerals Research Company purchased five gallons of commercial high purity fused silica slip from the Thermo Materials Corporation (TMC), Scottdale, Georgia. This slip was shipped to the Georgia Institute of Technology for measurement of slip characteristics prior to its being shipped to the Energy and Minerals Research Company. The slip was rolled in the five gallon carboy provided by TMC for 100 hours at 72 rpm prior to making measurements of percent solids content, apparent viscosity and pH. (The reasons for rolling the slip prior to measurement are given in Appendix A. Slip-Preparation.)

The pH was measured using a standard glass electrode pH meter at room temperature (22° C). The pH was measured without dilution of the slip. Solids content was measured by drying a 50 gram sample of slip to constant weight. The apparent viscosity was determined using a Brookfield Viscometer, model LVF, with a number two spindle. Measurements were made at 6, 12, 30 and 60 rpm. These data are shown in Table I.

The Thermo Materials Corporation provided information on chemical analysis and particle size distribution on each lot of slip. The chemical analysis was accomplished by Spectrochemical Laboratories, Pittsburgh, Pennsylvania, using a combination of wet-chemical and spectrographic techniques. An elemental and oxide analysis is shown in Table II with silica (SiO_2) reported by difference. The particle size distribution analysis was made by Micromeritics Instrument Corporation (MIC), Norcross, Georgia using the MIC Sedigraph 5000 Instrument. The particle size distribution obtained is shown in Figure 1.

After completing the measurements the 5 gallon container of slip was shipped to the Energy and Minerals Research Corporation.

B. Conventional Slip Casting of Fused Silica Plates

A second five gallon container of TMC 053182 HPB high purity fused silica slip was purchased under a parallel program (Contract N60921-82-C-0126) to prepare slip-cast fused silica plates. Fifty millimeter thick plaster plates were cast using a mixture of five parts U. S. Gypsum "Number One" pottery plaster and four parts water. The plaster plates were dried at 45° C for a minimum of three days prior to use. A frame consisting of four 25 millimeter by 12 millimeter methacrylic plastic strips were placed on the dried plaster plate to form a 25 millimeter deep cavity 160 by 160 millimeters. This cavity was filled to the top with fused silica slip, then covered with an additional sheet of plastic to prevent air drying of the top surface. The part was allowed to cast overnight then the four plastic strips were removed. The final thickness of this part was approximately 20 millimeters. After drying the average bulk density of small sections of plate was found to be 1874 kg/m^3.

Task II. Preparation of High Purity Fused Silica Grain

Fused silica cullet (broken tubing from chemical and optical ware lines) equivalent to General Electric 204 a type I* silica glass was ground in one gallon 85 percent alumina ball mills loaded with two kilograms of 85 percent alumina grinding media. A one kilogram charge of fused silica tubing was

*Type I silica glasses are produced from natural quartz by electrical fusion under vacuum or an inert atmosphere.

placed in the mill and ground for ten minutes. The material was then dry screened into fractions between -20 mesh (840 μm) and +270 mesh (53 μm). This process was repeated until three kilograms of materials of the desired fractional size were obtained.

Three high purity fused silica grain distributions were supplied to the Energy and Minerals Research Company: (1) -10 +20 mesh, (2000 to 840 μm diameters); (2) -20 +50 mesh, (840 to 297 μm); and (3) a blend conforming as closely as possible to curve number 2 in Figure 2. This distribution was a blend of: 24.5 percent, -25 +45 mesh, (710 to 350 μm); 32.7 percent, -45 +80 mesh, (350 to 177 μm); 26.5 percent, -80 +170 mesh, (177 to 88 μm); and 16.3 percent, -170 +270 mesh, (88 to 53 μm). These blends were shipped to the Energy and Minerals Research Company on 20 October 1982.

Task III. Sintering of Conventionally Slip-Cast and Ultrasonically Assisted Slip-Cast Fused Silica Plates

A. Sintering of Conventionally Slip-Cast Fused Silica Plates

The conventionally slip-cast fused silica plates were sintered in a rotating hearth, electrically resistance heated kiln. The plates were placed on a layer of alumino-silicate fiber which was in turn placed on a foamed fused silica plate located at mid-height inside the kiln. Six plates were arranged in a circular pattern and the kiln hearth was rotated at 4 rpm. The heating elements are located in one side of the furnace so that by rotating the circularly arranged specimens each plate would "see" an equal amount of radiation from the heating elements during the firing.

The procedure followed in firing the silica plates was to raise the temperature to 1225 K in 15 hours (overnight). The temperature was raised to the desired firing temperature in 90 minutes and was held at temperature for the desired firing interval. The electrical power was then shut-off and the kiln allowed to cool overnight before opening. The initial firing was at 1463 K for 315 minutes. After firing one plate was broken into several pieces and the bulk density determined by boiling in water, cooling, weighing wet, suspended and dry. These weights were then used to calculate the bulk density as described in ASTM # C-373. The average bulk density calculated for this firing was 1943 kg/m^3, slightly below the desired 1950 kg/m^3.

A second firing was made at 1469 K for 340 minutes. One plate was broken up and measured for bulk density following the procedures of ASTM C-373. The average bulk density for this plate was 1948 kg/m^3, hence all additional plates supplied to NSWC were fired at 1469 K for 340 minutes.

B. Sintering of Ultrasonically Assisted Slip-Cast Fused Silica Plates

Since the number of ultrasonically assisted slip-cast fused silica plates received from Energy and Minerals Research Company for sintering was limited, it was not considered advisable to break any of these plates for bulk density determinations. Two of the plates were broken as received. The first an aggregate cast plate made with commercial grade fused silica grain was broken into four nearly equal sections with one of the four sections retained by the Energy and Minerals Research Company. One of the five slip-cast plates received for sintering was broken into three pieces, one of which was a small corner piece. This small corner and a section broken from one of the three aggregate cast pieces were used to obtain dry bulk density values using a mercury volumeter. The aggregate cast piece had a dry bulk density of 1770 kg/m^3 and the slip-cast piece had a dry bulk density of 1874 kg/m^3.

A 19 millimeter diameter by 150 millimeter long cylindrical bar conventionally slip-cast was broken into three sections to be sintered along with the ultrasonically assisted slip-cast fused silica plates. This bar had a dry bulk density of 1870 kg/m^3. The initial firing was conducted on two of the three pieces of the broken slip-cast plate and the three aggregate cast pieces. This firing was at 1469° K for 340 minutes, the same conditions as used on the conventionally slip-cast plates described in Section A. Bulk density was calculated by the methods of ASTM C-373 and the results are shown in Table III. Since these values were considerably below the desired 1950 kg/m^3, a second firing was conducted on the same plates at 1494° K for 160 minutes. These values are also shown in Table III.

As can be seen in the table the 19 millimeter diameter conventionally cast bars are much denser than the desired 1950 kg/m^3 after the second sintering while none of the three ultrasonically assisted slip-cast parts have reached the desired density. The aggregate cast parts are much lower in density due to the low dry density. X-ray diffraction analysis on the small slip-cast piece indicated a cristobalite content of 0.7 percent. Additional sintering of these parts would result in a rapid increase in the cristobalite content. For slip-cast material of this density it is desirable to have less than one percent cristobalite in the final fired part, therefore, further sintering of these slip-cast parts was not considered advisable. The sintered parts were shipped to Energy and Minerals Research Company.

TABLE I

SLIP PROPERTIES FOR THERMO MATERIALS
HIGH PURITY FUSED SILICA SLIP

		053182-HP-B
Solid Content (%)		82.7
pH @ 20° C		4.4
Viscosity @ 20° C (centipoise)		
	@ 6 rpm	185
	12 rpm	150
	30 rpm	133
	60 rpm	121
Mean Particle Size (μm)		7.8
% of Particles < 2 μm		23

TABLE II

CHEMICAL COMPOSITION OF THERMO MATERIALS
HIGH PURITY FUSED SILICA SLIP

	053182-HP-B
	Weight Percent
Al_2O_3	0.23
TiO_2	0.002
Fe_2O_3	0.003
MgO	0.007
CaO	0.001
CoO	0.001*
Cr_2O_3	0.001
SiO_2	99.76
	Parts Per Million
Na	10*
K	10*
Li	10*

*Not detected. The number indicates the minimum limit of detection.

TABLE III

EFFECT OF SINTERING TEMPERATURE-TIME ON BULK DENSITY OF CONVENTIONALLY SLIP-CAST AND ULTRASONICALLY ASSISTED SLIP-CAST HARDWARE

Specimen	Sintered at 1469^{o} K - 340 minutes (Bulk Density, kg/m^3)	Sintered at 1494^{o} K - 160 minutes (Bulk Density, kg/m^3)
Conventionally Slip-Cast Plate		
Specimen 1	1947	-
Specimen 2	1949	-
Specimen 3	1947	-
Conventionally Slip-Cast Bar		
Specimen 1	1917	1967
Specimen 2	1918	1971
Specimen 3	1922	1973
Ultrasonically Assisted Aggregate Cast Plate		
Specimen 1*	1805	1819
Specimen 2	1812	1827
Specimen 3	1804	1810
Ultrasonically Assisted Slip-Cast Plate		
Specimen 1*	1922	1943
Specimen 2	1907	1934
Specimen 3	-	1919

*Specimens used for dry bulk density measurements.

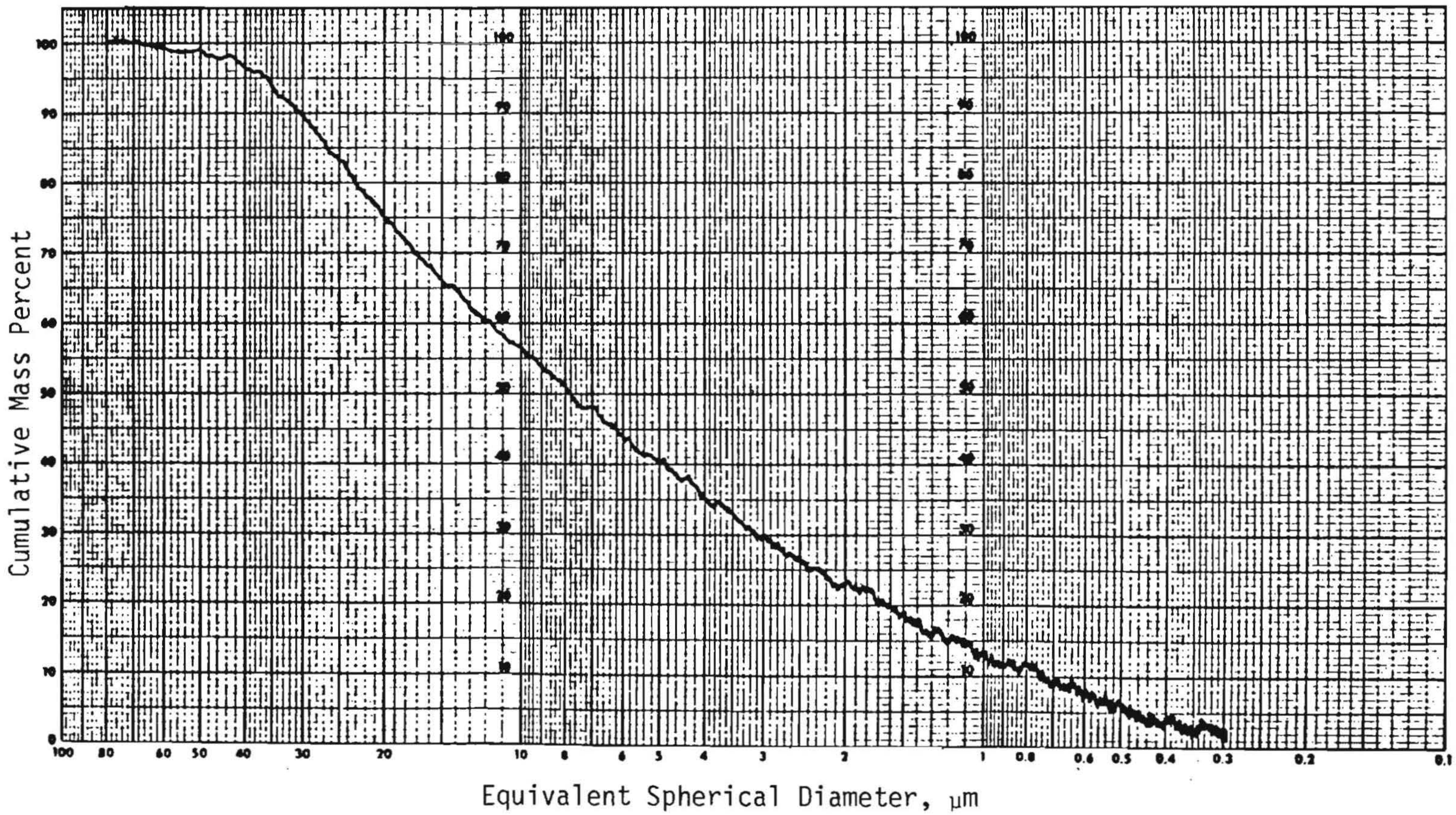

Figure 1. Particle Size Distribution of High Purity Fused Silica Slip 053182-B-HP

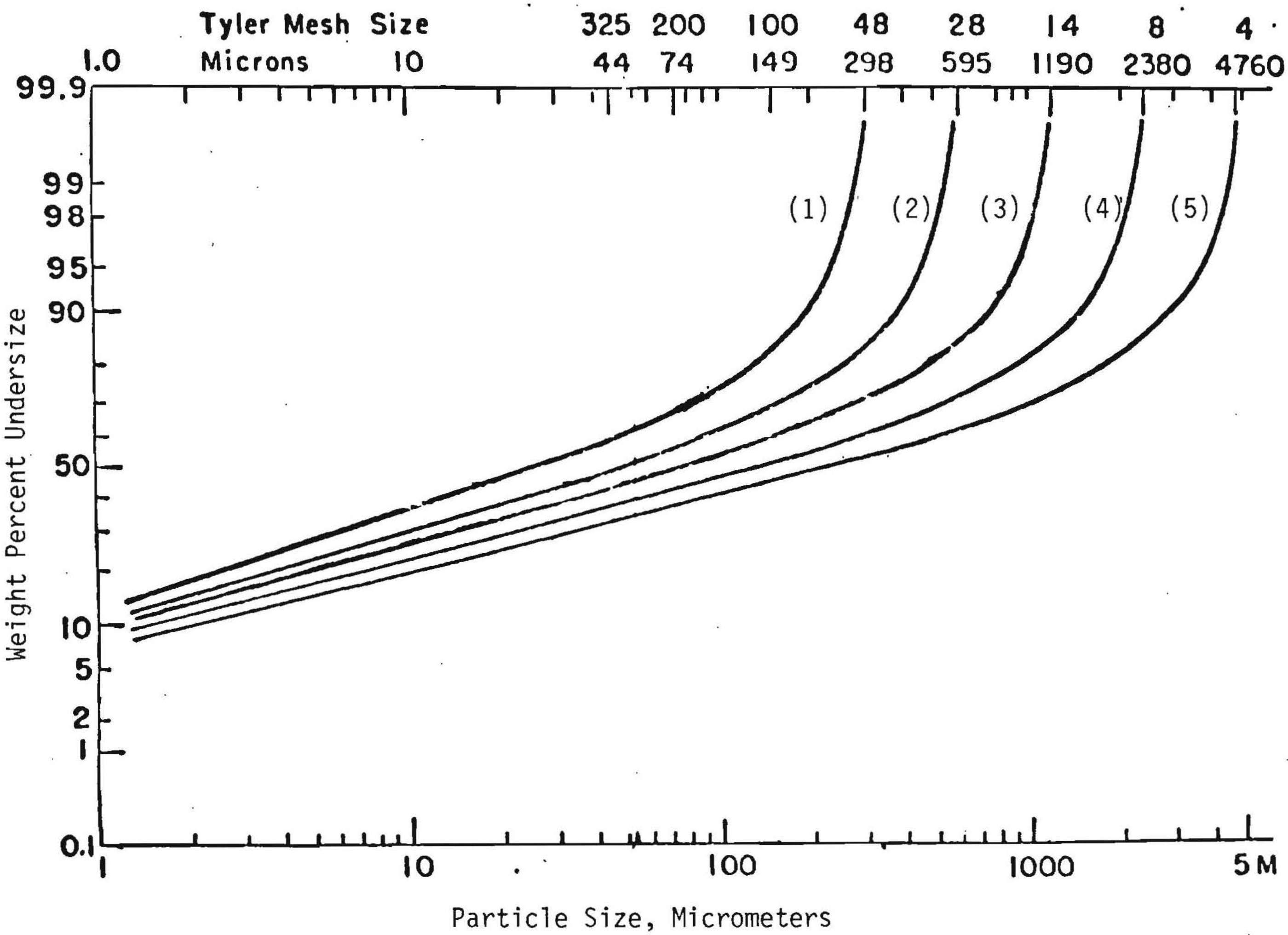

Figure 2. Continuous Particle Size Distribution Curves for Maximum Density (After Furnas)

(R. P. Helich, G. Maczura and F. J. Rohr, Alcoa Laboratories)

APPENDIX A

HIGH-PURITY FUSED SILICA SLIP PREPARATION PRIOR TO SLIP-CASTING IN PLASTER MOLDS

Freshly milled fused silica slip undergoes an "aging process" whereby the rheology of the slip and the properties of slip-cast hardware (as-cast density and ability to dry without cracking) continually change for a period of approximately 90 days. These changes are irreversible and once the "aging process" stops the slip can be used indefinitely without any further changes in the properties of cast hardware, provided that the slip is properly stored to prevent loss of water.

Pivinskii 1/ attributes this aging to a breakdown or shearing of the bonded water surrounding each individual particle in the slip. He further points out that large shear forces remove a portion of the bonded water, rapidly resulting in a change in rheology from dilatant to Newtonian to thixatropic.

Am empirical study was conducted at Georgia Tech 2/ in which fused silica slip was subjected to shear forces by rolling the container at peripheral surface speeds greater than one meter per second. Changes in pH and viscosity were monitored after different periods of time. The rheology of the slip checked in this study changed from dilatant to Newtonian and the pH stabilized after 48 hours of rolling. With continued rolling for periods up to 150 hours no further changes occurred in either pH or viscosity. To be certain that the slip has stabilized it is now standard practice to roll high purity fused silica slips for 100 hours, in the container in which it is received, at peripheral surface speeds of one meter per second. For the standard 19 liter polyethylene carboy this translates to 72 rpm. An additional advantage to this treatment is that it stabilizes the particles in suspension so that they do not settle out. A container of this slip will show no sediment after sitting undisturbed for a period of eight hours. However, it is recommended that slips be rolled at peripheral speeds of the order of 0.2 m/s during periods in which it will be used to keep it totally suspended. Agitation is not necessary for periods of casting up to eight hours. When the slip is not in use it may be stored without agitation. After long periods of storage it should be rolled at 0.2 m/s for 48 hours before use.

1/ Pivinskii, Yu E., "Processes in Producing Clinker and Casting Quartz Ceramics," Ogneupory, No. 7, pp 49-57, July 1971.

2/ J. N. Harris, "Effect of Rolling Velocity and Time on High Purity Fused Silica Slip Viscosity and Properties of Slip-Cast and Sintered Hardware," Final Report, prepared for Selenia Industrie Elettroniche Associate, SpA, Rome, Italy, September 1979.